Experimental part for the evaluation of the influence of the post-curing temperatures on the surface morphology and the suitability as a filler component in dental compositions of torus-shaped SiO_2 particles which were obtained by spray drying.

In order to reproduce the experimental results on which the patent DE10253481 is based, an aqueous SiO_2 nanosol (Ludox AS 40, average particle size 15 nm) was sprayed using the spray dryer "Mobile Minor 2000" of the company Niro A/S. The apparatus parameters described before (nozzle, air temperature, solid content, spraying pressure and flow rate) were used in doing so. As can be seen in the REM-pictures, the torus shape could be reproduced well (see REM pictures "ungetempertes Ausgangsmaterial " (not post-cured starting material)) Subsequently, the starting material was post-cured at 400, 600, 800, 850 and 900°C. The microscope pictures by means of high resolution REM show unambiguously that no effect compared with the starting material can be recognised on the surface at temperatures of 400-600°C. At 900°C, a fusing together of the particles becomes unambiguously visible. At temperatures up to 900°C, the particles can be deagglomerated without problems before or after the silanization. At a temperature of 1000°C, the particles fuse together, so that any deagglomeration was no more possible.

A first hint with respect to a modified surface morphology of the particles could be observed in the wet silanization of the powder batches. Whereas those powders which had been post-cured at 400 or 600°C, respectively, yielded a rather highly viscous silanization slurry, the powder at 800°C showed already a lower viscosity This viscosity became even less viscous when the particles were silanized at 850 or 900°C, respectively

In order to characterise the particles with regard to their utilisation as a main filler component for light-curing dental filling composites, a test series of experimental materials were produced and characterised, which were based on a 40% sol of SiO_2 particles with a primary article size of 15 nm in a dimethylacrylate mixture BisGMA:UDMA:TEDMA of 5:3:2 As an initiator system, camphorquinone/DMABE was used again.

In order to provide comparability with respect to the suitability as torus particles, the consistency of the resulting composite pastes was kept constant The same was a standard consistency of dental filling materials

In order to characterise the light-curing composites, the proportion of torus particles which could be incorporated maintaining the consistency which is aimed at, the overall filler content, the flexural strength (according to ISO 4049), the flexural elasticity module and the polymerisation shrinking accompanying the overall filler content (according to "curing contraction of composites and glass-ionomer cements, A.J. Feilzer, A J. DeGee, C.L Davidsson, Journal of Prosthetic dentistry, Vol 59, Nr 3, p. 297-300) were determined The results are compiled in the table below

Sintering temperature	400°C	600°C	800°C	850°C	900°C
Proportion of 40% SiO_2 sol [weight-%]	47,2	46,3	44,7	41,7	37,9
Proportion of silanized torus particles [weight-%]	52,8	53,7	55,3	58,3	62,1
Overall filler content [weight-%]	71,7	72,2	73,2	75,0	77,3
Flexural strength [Mpa]	108	126	136	136	138
Flexural elasticity module [Mpa}	9320	9900	11310	11410	11820
Volume shrinking [Vol-%]	2,84	2,8	2,66	2,51	2,29

From the proportions of torus particles that can be incorporated into the composite, it can be recognised unambiguously that a smooth particle surface, like that achieved in the sintering temperature range of 800 – 900°C, has a positive effect on the physical properties of the filler composites. Due to the smaller wetting effects between matrix and torus, the overall filler content can be increased for more than 5 weight-% compared to particles which were sintered at low temperatures Expressed otherwise, the proportion of the organic crosslinker matrix can be lowered for more than 15 weight-% (expressed as percentage of that matrix) via this step of the selection of the appropriate post-curing temperature This is accompanied by a significant increase of flexural strength and elasticity module Further, the shrinking through the curing can be lowered for 0,5 Vol-%.

Such an interrelationship (between sintering temperature and overall filler content) could not have been expected, it is completely new The increase of the filler content is the objective of every development of new dental composites Seen before this background, an increase of the filler content for 5 weight-% and accompanying decrease of the material proportion, as well as improvement of the relevant parameters,i s sheerly sensational

Cuxhaven, October 21 2008

Dr. Reinhard Maletz